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**Nuclear fuel technology — Alpha  
spectrometry —**

**Part 3:  
Determination of uranium 232 in uranium  
and its compounds**

*Technologie du combustible nucléaire — Spectrométrie alpha —  
Partie 3: Détermination de l'uranium 232 dans l'uranium et ses  
composés*



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## Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 21847-3 was prepared by Technical Committee ISO/TC 85, *Nuclear energy*, Subcommittee SC 5, *Nuclear fuel technology*.

ISO 21847 consists of the following parts, under the general title *Nuclear fuel technology — Alpha spectrometry*:

- *Part 1: Determination of neptunium in uranium and its compounds*
- *Part 2: Determination of plutonium in uranium and its compounds*
- *Part 3: Determination of uranium 232 in uranium and its compounds*

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# Nuclear fuel technology — Alpha spectrometry —

## Part 3:

# Determination of uranium 232 in uranium and its compounds

## 1 Scope

This part of ISO 21847 describes a method for determining trace amounts of  $^{232}\text{U}$  in uranium hexafluoride, uranium oxides or uranyl nitrate.

## 2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

## 3 Principle

Undesirable elements (e.g. Th) are removed by liquid-liquid extraction.

Sources are prepared by electro-deposition and  $^{232}\text{U}$  is assayed by alpha spectrometry.

## 4 Reagents

Use reagents of recognized analytical grade.

**4.1 Water**, at least quality 1 as specified in ISO 3696.

**4.2 Nitric acid**, concentrated,  $c(\text{HNO}_3) = 15,8 \text{ mol/l}$ .

**4.3 Nitric acid**,  $c(\text{HNO}_3) = 7 \text{ mol/l}$ .

**4.4 Nitric acid**,  $c(\text{HNO}_3) = 2 \text{ mol/l}$ .

**4.5 Nitric acid**, dilute,  $c(\text{HNO}_3) = 0,1 \text{ mol/l}$ .

**4.6 Thenoyltrifluoroacetone**, (TTA;  $\text{C}_8\text{H}_5\text{F}_3\text{O}_2\text{S}$ ) solution in xylene,  $c(\text{C}_8\text{H}_5\text{F}_3\text{O}_2\text{S}) = 0,25 \text{ mol/l}$ .

Prepare the solution weekly.

**4.7 Ammonium chloride solution**, saturated, ( $\text{NH}_4\text{Cl}$ ).

**4.8 Methyl red solution**.

4.9 **Sodium hydroxide solution**,  $c(\text{NaOH}) = 5 \text{ mol/l}$ .

4.10 **Hydrochloric acid**, dilute,  $c(\text{HCl}) = 1 \text{ mol/l}$ .

4.11 **Gas mixture**, with a volume fraction of 90 % argon and a volume fraction of 10 % methane.

## 5 Apparatus

5.1 **Standard laboratory equipment.**

5.2 **Electro-deposition device.**

5.3 **Frisch grid ionization chamber**, with argon-methane gas mixture, or **semiconductor detector**.

5.4 **Multichannel amplitude analyser.**

5.5 **Stainless steel disks**, approx. 60 mm diameter, or **watch glasses**.

## 6 Preparation of test sample

6.1 Hydrolyse the  $\text{UF}_6$  sample. Prepare uranyl solution.

6.2 Dissolve the oxide sample in concentrated nitric acid (4.2) to obtain uranium nitrate solution.

NOTE Because the method is applicable to uranium compounds of nuclear purity, other compounds can require prior extraction by tributyl phosphate, TBP, in nitric acid (4.3) to obtain purified uranium.

## 7 Procedure

### 7.1 Preparation of test solution

7.1.1 Place a test portion containing about 1 g of uranium in a beaker.

7.1.2 Add 10 ml of concentrated nitric acid (4.2).

7.1.3 Dry.

7.1.4 Repeat three times (for  $\text{UF}_6$  hydrolyzates) the operations 7.1.1 to 7.1.3.

7.1.5 Evaporate dry, then dissolve in 10 ml of dilute nitric acid (4.5).

### 7.2 Extraction

7.2.1 Pour the solution into a separatory funnel; rinse the beaker three times with 1 ml of dilute nitric acid (4.5).

7.2.2 Add the rinse solutions to the test solution.

7.2.3 Add 5 ml of TTA (4.6) and agitate mechanically for 10 min.

7.2.4 Allow the phases to separate.

7.2.5 Recover the aqueous phase.

- 7.2.6** Add 5 ml of TTA (4.6) and agitate mechanically for 10 min.
- 7.2.7** Allow the phases to separate.
- 7.2.8** Recover the aqueous phase in a 100 ml flask, which is designated Flask A.
- 7.2.9** Adjust to 100 ml with dilute nitric acid (4.5).

### 7.3 Preparation of source sample

- 7.3.1** Place about 25 ml of saturated ammonium chloride (4.7) in the assembled electrolysis tank of the electro deposition device (5.2).
- 7.3.2** Add the assay sample. Flask A (7.2.8) contains between 100 µg and 400 µg of uranium.
- 7.3.3** Add 2 drops of methyl red solution (4.8).
- 7.3.4** Shake.
- 7.3.5** Add 1 drop of sodium hydroxide solution (4.9) to alkalize the medium.
- 7.3.6** Add dilute hydrochloric acid (4.10) until the solution just becomes acid (methyl red changes colour).
- 7.3.7** Centre the anode and immerse it in solution about 25 mm from the cathode.
- 7.3.8** Perform electrolysis with a current of about 4 A for 10 min.
- 7.3.9** After 10 min, add 2 drops of sodium hydroxide solution (4.9) before switching off the power (to prevent the deposit from dissolving), remove the cathode, rinse it in water and dry with compressed air.

NOTE The electro-deposition parameters depend on the geometry of the electrolysis cell.

**7.3.10** Decontaminate the anode and the electrolysis cell using a solution of a cleaning product adapted to the radioactive decontamination of surfaces, then clean in hot, dilute nitric acid (4.4).

### 7.4 Measurement

- 7.4.1** Place the source in the Frisch grid ionization chamber (or semiconductor detector) (5.3) connected to a multichannel amplitude selector (5.4).
- 7.4.2** Create a vacuum in the chamber, then fill it with the gas mixture (4.11).
- 7.4.3** Set the multichannel analyser to the 4 MeV to 6 MeV energy band.

NOTE The counting time depends on the source activity and the detection system.

**7.4.4** Periodically perform background counts for the same time on a stainless steel disk without any deposit.

## 8 Expression of results

After acquiring the spectrum, identify and integrate the  $^{232}\text{U}$  peak at 5,320 MeV and the  $^{238}\text{U}$  peak at 4,196 MeV.

Record the calculated activities for  $^{232}\text{U}$  ( $A_{232\text{U}}$ ) and  $^{238}\text{U}$  ( $A_{238\text{U}}$ ).

Calculate the mass fraction,  $w_{232\text{U}}$  of  $^{232}\text{U}$  relative to the total uranium as given in Equation (1):

$$w_{232\text{U}} = \frac{A_{232\text{U}}}{A_{238\text{U}}} \cdot \frac{a_{238\text{U}}}{a_{232\text{U}}} \cdot \left(1 - \frac{w_{\text{min}}}{100}\right) \quad (1)$$

where

- $A_{238\text{U}}$  is the calculated activity of  $^{238}\text{U}$ , expressed in becquerels;
- $A_{232\text{U}}$  is the calculated activity  $^{232}\text{U}$ , expressed in becquerels;
- $a_{238\text{U}}$  is the massic activity of  $^{238}\text{U}$ , expressed in becquerels/gram (equal to  $1,25 \times 10^4$  Bq/g);
- $a_{232\text{U}}$  is the massic activity of  $^{232}\text{U}$ , expressed in becquerels/gram (equal to  $7,9 \times 10^{11}$  Bq/g);
- $w_{\text{min}}$  is the sum of the mass fractions for the minor isotopes of uranium, expressed as a percent of the total.

## 9 Precision

Reproducibility standard deviation is 0,03 ng  $^{232}\text{U/g}$  U for 19 measurements with a 10-h counting period at the level of 1,22 ng  $^{232}\text{U/g}$  U. Reproducibility results apply to enrichments up to 5 %.

NOTE This value was obtained by imposing strict requirements on the cleanliness of the work environment and measurement system.

## 10 Test report

The test report shall include the following information:

- a) identification of sample;
- b) method used by reference to this part of ISO 21847;
- c) results and the form in which they are expressed;
- d) any unusual features noted during the test;
- e) any operations not included in this part of ISO 21847, or regarded as optional.



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